1. TITLE OF THE THESIS:

“APPLICATION OF NANO MAGNETITE LOADED COPOLYMERIC HYDROGELS IN THE EFFECTIVE REMOVAL OF TOXIC METAL IONS FROM EFFLUENT WATER”

2. INTRODUCTION:

The presence of heavy metals in the wastewater has been a matter of considerable concern in the recent years because of their toxic properties. The intensification of industrial activity during recent years is greatly contributing to the increase of heavy metals in the environment, mainly in the aquatic system [1]. Wastewater containing heavy metals is a serious environmental problem because they do not undergo biodegradation and are accumulated into the organism entering into the food chains [2]. Therefore, it is important to take effective precautions to prevent our natural water resources by controlling emission of toxic heavy metals into the environment.

The presence of heavy metals in natural water results from two independent factors. The first involving the weathering of soil and rocks with its products are being transported by air and water and the second involving a variety of anthropogenic activities that have potential impact on human health [3]. The increasing levels of toxic metals that are discharged to the environment as industrial wastes represent a serious threat to human health, living resources and ecological systems. The industries responsible for the discharge of wastewater containing metals are mining and mineral processing, pigment manufacturing, painting and photographic industries, metal-working and finishing processes, energy and fuel production, fertilizer and pesticide industry, leatherworking, electric appliances manufacturing, metal surface treating aero space and atomic energy installation etc.

If the metal ions are ingested beyond the permitted concentration, they can cause serious health disorders [4]. Therefore it is necessary to develop the methods that allow one to detect, quantify and remove these heavy metals from the effluent waters. Among these heavy metal ions Cd, Zn, Hg, Pb, Cr, Cu, As etc. gain importance due to their high toxic nature even at very low concentrations. In recent years a general environmental awareness has developed that is related to the actual knowledge and control of water pollution.

Existence of heavy metal pollutants in water results in ecological problems even at very low concentrations which increased the need for materials that can provide efficient complexing potential towards these metal ions [5]. A number of Physical, chemical and biological technologies have been applied for removal of heavy metal ions from aqueous solutions. The common reported methods for decontamination of water are filtration, chemical precipitation, coagulation, solvent extraction, electrolysis, ion exchange, membrane process and adsorption [6-10]. However, most of these methods might not be efficient in removing heavy metals at very low concentrations, and could be relatively expensive [11, 12]. Therefore, the search for newer treatment technologies for removal of toxic metals from wastewaters has directed attention to sorption onto synthetic magnetic nano particles loaded cation exchangers (ie. copolymeric hydrogels and resins).

In the present synopsis the adsorbents chosen for the removal of toxic metal ions are magnetic hydrogels composed of co-polymers of acrylamide/acrylonitrile with acids and some cationic resins magnetized insitu.

Hydrogels:

Hydrogels are three-dimensional high-molecular weight networks composed of a polymer backbone, water and a cross linking agent. These are polymeric materials that do not dissolve in water at physiological temperature and pH [13-16]. They swell considerably in an aqueous medium [17] and demonstrate extraordinary capacity for imbibing water into the network structure [18].

The water sorption property of hydrogels accounts for a great number of biomedical and technological applications such as agricultural uses, controlled drug release, artificial muscles, membranes and water treatment industry [19-21]. Recently, there is a great deal of interest concerning the production of nano particles in the hydrogel networks since they have enormous valuable applications [22]. Hydrogels have a wide range of applications as controlled drug delivery vehicles, in tissue regeneration, as artificial organs in biotechnology, sensors, in separation and purification processes, and the recognition of certain molecules, bio-molecules and proteins [23, 24]. Several polysaccharides-based hydrogels have been employed to adsorb chemicals in wastewater treatment processes and have attracted particular interest [25].

Magnetic nano particles of iron oxide:

Magnetic nanoparticles (MNPs) have been the subject of intense research over the past decade because of their potential applications as electronic, photonic, magnetic and biomedical materials. Magnetic separation is an alternative approach which exhibits promising solid-liquid phase separation of various metals. Because of extremely small size and high surface to volume ratio results in super adsorption kinetics for various metal ions in aqueous solutions. Nano-sized magnetite (Fe_3O_4) and other nanomaterials are beginning to show many advantages, especially in the environmental science. Magnetic nanoparticles offer a unique and useful magnetic property as super paramagnetism, which arises from the nano-structured particles of Fe_3O_4 [26]. Nano particles of iron-oxide with high surface energy and surface area have strong adsorption capability, high magnetic properties and involve easy and rapid separation of adsorbent from solution with respect to many inorganic ions and organic matters. The combined technique of sorption and magnetic separation holds the advantageous flexibility, ecofriendly characteristic and recovery of heavy metals is very important and inspiring. In order to improve the adsorption capacity of magnetic nanoparticles, the active functionalization by various active polymers/co-polymers which can impart surface reactivity towards various metal ions is needed.

3. A BRIEF REWIEW OF THE WORK ALREADY DONE IN THE FIELD:

Y. Zheng et al. [27] studied the removal of Cu^{2+} onto starch-g-poly (acrylic acid) / sodium humate hydrogels by batch method and found that these hydrogels hold great potential to remove Cu^{2+} from aqueous solution. H. Kasgoz et al. [28] employed hydrogel with acid groups for removal of copper (II) and lead (II) ions from aqueous solution. Adsorption of heavy metal ion onto electron beam grafted copolymer hydrogel from maleic acid, poly (vinyl alcohol) and methylene bisacrylamide was carried out by P. Chowdhury et al. [29]. This synthesized hydrogels were act as cation exchanger with high exchange capacity.A. shanmugapriya et al. [30] carried out the biosorption of Cu (II) and Cr (VI) ions from wastewater by chitosan-g-acrylic acid. This adsorbent was found to be favorable in the removal of metal ions from the wastewater. Significant result was found for the industrial application. Removal of metal ions (Co (II), Ni (II), and Cu (II)) from aqueous solutions by magnetic hydrogel nanocomposites was reported by N.N. Reddy [31].

Poly (sodium acrylate) hydrogels as potential pH-sensitive sorbents for the removal of model organic and inorganic pollutants from water were reported by V. Bekiari et al. [32]. It was found that these hydrogels are efficient sorbents for both pollutants. Moreover, the adsorption capacity of these hydrogels was much larger towards Cu^{2+} ions, as compared to crystal violet. This is attributed to the formation of coordination complex in the first case, whereas electrostatic binding is the major driving force in the case of crystal violet. U. Yildiz et al. [33] used novel pH-sensitive hydrogels for removal of heavy metal ions (Cu^{2+}, Cd^{2+}, Ni^{2+} and Zn^{2+}). The maximum binding capacity of the hydrogels for different metal ions was in the order of Cu^{2+}>Ni^{2+}>Zn^{2+}>Cd^{2+}. N. Sahiner [34] reported hydrogels of versatile size and architecture for removal of dyes and arsenic metal ions. The hydrogels prepared in this study were proven to be very useful for environmental applications, such as removal of dyes and toxic metal ions from an aqueous media. Removal of lead ions from aqueous solution using (sodium alginate / itaconic acid) hydrogel carried out by G.A. Mahmoud et al. [35]. The results of above study demonstrated that this hydrogel may be an extremely viable adsorbent in the treatment of water and industrial wastewater contaminated with Pb^{2+}.

4. OBJECTIVES:

The proposed synopsis includes the following objectives.

1. Synthesis of various copolymeric hydrogels.
2. Insitu magnetization of synthetic copolymers.
3. Characterization of magnetite loaded hydrogels and resins by FTIR, TEM, XRD, AFM and SEM analysis.
4. Study of removal of toxic metal ions such as copper, lead, cadmium, zinc, mercury, arsenic, chromium, iron etc.
5. Study of dynamics of adsorption of metal ions onto magnetite nano particles loaded copolymeric hydrogels and resins and to observe the influence of various experimental conditions on the percentage removal of metal ions.
6. Study of different factors like pH, time, temperature, adsorbent dose etc.
7. Study of kinetic and thermodynamics of metal ion sorption.
8. Study of adsorption by fixed-bed adsorption column method.

5. NOTE WORTHY CONTRIBUTION IN THE FIELD OF PROPOSED WORK:

Removal of Co(II), Cu(II) and Pb(II) ions by polymer based 2-hydroxy ethyl methacrylate: thermodynamics and desorption studies was reported by Moradi etal [36]. The feasibility of cross-linked maleic anhydride-heptene-1-styrene terpolymer containing various functional groups as adsorbent for removing uranyl ions from aqueous solution was investigated by O.H. Akperov et al. [37]. A study on the removal of Cu (II) ions with poly(N-isopropylacrylamide-co-methacrylic acid) micro/nanoparticles was carried out by Wu etal [38]. Adsorption of lead on to poly (acrylamide-co-maleic acid) based hydrogel composites were reported by Akkaya etal [39]. The effluence of various experimental parameters was studied. Adsorption of Cu (II) ions onto poly (acrylic acid-co-acrylamide) hydrogels was studied by Orozco-Guareno etal [40]. Bajpai etal investigated removal of Cu(II) from aqueous solutions using magnetic exchanger resin [41]. Ion exchanger resin Amberjet 1200H was applied as adsorbent for removal of Cd (II) ions from aqueous solution by Bai etal [42]. Adsorption behavior of cadmium (II) from aqueous solution by D152 resin was reported by Xiong etal [43]. Adsorption behavior of gel-type weak acid resin (110 – H) for $Pb^{2+}$was studied by Xiong etal [44]. Diniz etal reported effect of pH on the adsorption of selected heavy metal ions from concentrated chloride solutions by the chelating resin dowex M – 4195 [45].

6. PROPOSED METHODOLOGY:

The methodology may be described as follows.

1. Synthesis of magnetic copolymeric hydrogels:

- To a mixture of monomers, the cross linker and initiator will be added and heated at 70^{o}C in an electric oven for 1 h.
- The copolymer so formed will be washed with distilled water and cut into small pieces of uniform sizes.
- For in-situ magnetization, these pieces will be equilibrated in an aqueous solution of ferrous chloride and ferric chloride for 24 hr followed by adding concentrated ammonia solution.

- The Magnetic copolymeric hydrogel will then be washed thoroughly with distilled water, dried and crushed into a fine powder.

2. Characterization of magnetic copolymeric hydrogels:

The magnetic copolymeric hydrogels will be characterized by

- **Fourier Transform Infrared Spectroscopy (FTIR) spectral analysis** – to determine the presence of specific functional groups and coating of magnetic nano particles in copolymeric hydrogels.
- **Transmission Electron Microscopy (TEM) –** to determine the size and shape of magnetic nano particles.
- **X-ray Diffraction Analysis (XRD) –** to evaluate the phase identification of the magnetic nano particles.
- **Atomic Force Microscopy (AFM)** – to investigate the morphology and size of magnetic nano particles.
- **Scanning Electron Microscopy (SEM)** – to study the morphology of the surface of magnetic copolymeric hydrogels.
- **X-ray Photoelectron Spectroscopy (XPS) Analysis –** to determine the oxidation state of iron and ratio of Fe^{3+} and Fe^{2+}.

3. Adsorbate:

- Various metal ion solutions such as Cu (II), Pb (II), Cd (II), Zn (II), Hg (II), As (V), Fe (II), Cr (VI) etc will be used as adsorbates.

4. Mode of adsorption:

1. *Batch method:*

- The adsorption experiments will be carried out by batch method by varying pH, contact time, adsorbent dose, adsorbate concentration and temperature.
- The adsorption experiments will be conducted by shaking a fixed volume of adsorbate with a definite amount of magnetic copolymeric hydrogel in aqueous medium.

- The amount of metal ions present in solution (before and after adsorption) will be determined by Atomic Absorption Spectrometer.
- The sorption degree (percentage removal) and sorption capacity of the sorbent will be calculated by the following equations:

$$\text{Sorption degree} = \frac{(C_o - C_e)}{C_o} \quad X\ 100\ \%$$

$$\text{Sorption capacity} = \frac{(C_o - C_e)\ X\ V}{m}$$

Where C_o and C_e (mg dm^{-3}) are initial and equilibrium concentrations of metal ions solutions respectively, V (L) is the volume of the metal ion solution subjected to sorption and m (g) is the weight of sorbent.

The following studies will be carried out in this part:

a. Adsorption isotherms and adsorption coefficient:

- In order to know the nature of adsorption process Langmuir, Freundlich and Temkin adsorption isotherm equations will be considered and by the shape of adsorption isotherms various parameters will be determined.

b. Effect of pH:

- The pH of the aqueous solution is an important controlling parameter in the adsorption process and influenced the removal efficiency of the metal ions.
- Hence in order to study the effect of pH, different adsorption experiments will be carried out by varying initial pH of solution in the range from 2 to 8.

c. Effect of temperature:

- Temperature plays an important role on the process of adsorption.

- In general the number of active sites generated on the adsorbent and the binding forces of adsorption are largely affected by the temperature and, therefore, the adsorption is directly affected. So it is necessary to study the effect of temperature on the adsorption of metal ions.

d. Effect of time:

- The effect of time on the removal of heavy metal ions will be studied by performing the adsorption experimental at different time intervals for maximum removal of heavy metal ions.

e. *Effect of adsorbent dosages*:

- The dependence of metal ion sorption on adsorbent dose will be studied by varying the amount of adsorbent to optimize adsorbent dose at which maximum removal of toxic metal ions will occur.

f. Adsorption kinetic modeling:

- The kinetic investigations will be carried out to measure the adsorption rate under various experimental conditions such as pH, temperature and equilibrium time.
- Kinetic parameters, rate constants, equilibrium sorption capacities and related correlation coefficients for each kinetic model will be calculated and discussed.
- The kinetics of the sorption will be analyzed using the following kinetic models:

1. First order reversible model
2. Second-order kinetic model
3. Pseudo-first-order kinetic model
4. Pseudo-second-order kinetic model
5. Intra particle diffusion.

g. Thermodynamic study:

- Thermodynamic parameters such as the free energy (ΔG^0), enthalpy (ΔH^0) and entropy (ΔS^0) changes during adsorption will be evaluated to determine feasibility, spontaneity and exothermic/endothermic nature of adsorption.

2. *Fixed-bed adsorption column study:*

- Fixed-bed adsorption studies will be conducted in a column made of polyethylene at a constant temperature and pH.
- The column will be packed with different bed heights of magnetic hydrogel on a glass-wool support.
- A known concentration of metal ion solution will be allowed to pass through the bed at a constant flow rate in a down-flow manner.
- The metal ion solution will be then collected at different time intervals and the concentration of metal ions will be determined by Atomic Absorption Spectrometer.
- The important design parameters such as column bed height, flow rate of metal solution into column and initial concentration of metal solution will be investigated.
- The efficiency of the column was evaluated by determining breakthrough curves.

The following studies will be carried out in this part:

a. Effect of bed height:

- The effect of bed depth on the breakthrough curves will be investigated for beds with different heights at constant adsorbate feed flow rate and adsorbate inlet concentration.

b. Effect of flow rate:

- The effect of feed flow rate on the adsorption of metal ions on magnetic copolymeric hydrogels will be investigated by varying the feed flow rate with constant adsorbent bed height and inlet adsorbate concentration.

c. Effect of adsorbate inlet concentration:

- The effect of adsorbate metal ions concentration on the column performance will be studied by varying the inlet concentration with constant adsorbent bed height and feed flow rate.

d. Modeling of column dynamics:

- The sorption performance of the metal ions through the column will be analyzed by applying some of the models listed below.

1. Thomas model
2. Yoon-Nelson model
3. Adams-Bohart model
4. Wolborska model
5. BDST model.

7. EXPECTED OUTCOME OF THE PROPOSED WORK:

- The aim of the present research work is to assess the potential of the novel eco-friendly, non-toxic and bio-degradable magnetic copolymeric hydrogels in removing toxic heavy metal ions from aqueous solutions as well as industrial effluents.
- The proposed magnetic adsorbent based removal process is expected to be much cheaper, efficient and effective than other adsorbents and can be easily prepared and stored.
- The novelty of this work is that the loaded magnetic adsorbent can be desorbed in acidic medium and can be reused for further adsorption experiments.
- By this type of adsorption-desorption cycles, the columns may be designed for maximum removal of toxic heavy metal ions.
- The proposed magnetic hydrogels may be cost effective as well as efficient adsorbents for the removal of both organic and inorganic pollutants.

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9. PAPERS PUBLISHED/COMMUNICATED:

PAPER PUBLISHED:

1. "Kinetic and thermodynamic studies of Cd^{2+} adsorption onto super paramagnetic nano iron oxide loaded Poly (Acrylonitrile-co-Acrylic acid) Hydrogel**"**, ***Research on Chemical Intermediates (Springer)*** DOI No. 10.1007/s11164-013-1330-x.

PAPER COMMUNICATED:

1. "Kinetic and thermodynamic study of Zn^{2+} ions adsorption onto super paramagnetic Amberlite IR 120", ***Mechanics of Time Dependent Materials (Springer).*** Manuscript ID – MTDM-D-13-00046.

CONFERENCES ATTENDED:

1. **Oral presentation-** 2nd International Science Congress held on 8th-9th December, 2012, Organized by International Science Congress Association, at Vrindavan, Mathura, U.P., India. By Neeraj Sharma

   Title of Paper- "Synthesis and Efficiency of Magnetic Nanoparticles Loaded Poly (Acrylamide-co-Maleic acid) hydrogel in uptaking Copper Ions from Aqueous Solution".

2. **Poster Presentation-** International Conference on Chemistry and Materials: Prospects and Perspectives-2012, held on 14th-16th December, 2012, organized by Department of Applied Chemistry, School for Physical Sciences, Babasaheb Bhimrao Ambedkar University, Lucknow, U.P., India.

   Title of paper- "Kinetic and thermodynamic studies of Cd^{2+} adsorption onto super paramagnetic nano iron oxide loaded Poly (Acrylonitrile-co-Acrylic acid) Hydrogel.

WORKSHOPS ATTENDED:

1. Second School on Analytical Chemistry-2011 (SAC-2011), Organized by Association of Environmental Analytical Chemistry of India, C/o Analytical Chemistry Division, Bhabha Atomic Research Centre, Trombay, Mumbai, India, (21st-27th August, 2011) at National Centre for Compositional Characterization of Materials (NCCCM), Hyderabad, India.

2. National Workshop Cum Seminar on Advances in Electron Microscopy & Allied Fields, held on September 23rd-29th, 2011, organized by Department of

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